FUNDAMENTAL STUDIES OF NEURAL STIMULATING ELECTRODES

Eleventh Quarterly Report
Covering Period March 1, 1997 to May 28, 1997

CONTRACT NO. N01-NS-4-2310

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This QPR is being sent to you before it has been reviewed by the staff of the Neural Prosthesis Program.

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1. INTRODUCTION AND SUMMARY

This report describes work on NINDS Contract No. N01-NS-4-2310 during the period March 1, 1997 to May 28, 1997. As part of the Neural Prosthesis Program, the broad objectives of the present fundamental studies are: 1) to evaluate the electrochemical processes that occur at the electrode-electrolyte interface during pulsing regiments characteristic of neural prosthetic applications; 2) to establish charge injection limits of stimulation electrode materials which avoid irreversible electrochemical reactions; 3) to develop an *in vitro* method, which can be applied *in vivo*, for determining the electrochemical real area and stability of microelectrodes; 4) to develop new materials which can operate at high stimulation charge densities for microstimulation; and 5) to provide electrochemical and analytical support for other research activities in the Neural Prosthesis Program at NINDS.

During this reporting period, long term studies of U. Michigan ribbon-cable-probe No. 1, site 1 were continued. The apparent capacitance of the inactivated site, calculated from the current at 0.0 V vs. Ag|AgCl in a 200 V/s potential sweep, has been monitored for over 619 days of soaking in PBS. An abrupt but modest increase in the capacitance from 0.5 nF to ~2.5 nF was observed at day 91 of the soak test. On day 217, the electrode was inadvertently allowed to dry out. After the electrode was rehydrated, the apparent capacitance had decreased to a value closer to that observed before day 91. There has been no increase in C_{app}, similar to that observed at day 91, for the 402 days of soaking since the electrode was rehydrated at day 217.

This quarter we began an investigation to establish the reproducibility of the charge injection process for AIROF. The AIROF is produced by potential pulse activation in PBS between -0.70 and +0.80 V vs Ag|AgCl, using a halfwave of 10 seconds. The ability of AIROF to accommodate charge injection in the form of 200 μsec, biphasic, constant current pulses of equal cathodic and anodic magnitude for a sustained 3 minute period was examined. Results from the three constant current pulsing experiments performed to date indicate that as charge storage capacity increases, the charge injection limit decreases.

2. VOLTAMMETRIC STUDIES ON Ir MICROELECTRODES

During this quarter we continued studies of the long term stability of Ir sites on a probe with an integrated ribbon cable received from U. Michigan. Figure 2.1 is a diagrammatic representation of the probe showing the numbering scheme for the electrode sites. The probe has been under soak in phosphate buffered saline (PBS) for 619 days.

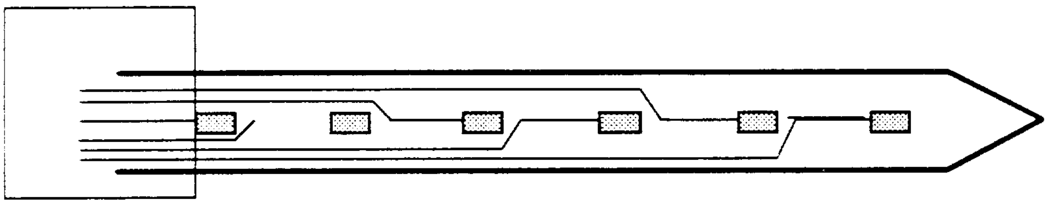

Figure 2.1 Diagrammatic representation of sites and site identification on U. Michigan ribbon-cable probe.

Measurements of the apparent capacitance of site 1 of the ribbon-cable-probe No. 1 were continued. Test procedures and results are detailed in previous Quarterly Progress Reports (Nos. 5-10). The apparent capacitance, C_{app}, at 200 V/s is plotted in Fig. 2-2 as a function of time for the study performed this quarter along with data from the 28 studies conducted during the previous six quarters. C_{app} is calculated by dividing the current at 0.0 V vs. Ag|AgCl by the scan rate, thus $C_{app} = Q/V = i/v$, where v is the scan rate.

We do not have a satisfactory explanation for the increase in apparent capacitance between days 91 and 220. The inadvertent drying out of the electrode at day 217 presumably caused the decrease in C_{app}. However, there has been no increase in C_{app} similar to that observed at day 91 for the 307 days of soaking since the electrode was rehydrated at day 217. This result, and the abruptness of the increase observed at day 91, suggests that the increase in C_{app} is not due to an ongoing degradation process at the electrode site.

A comparison of the voltammetric scans used in the calculation of C_{app} at 200 V/s for days 59, 160, 354, 517 and 619 is shown in Fig. 2-3. The 59, 354, 517 and 619 day scans are not significantly different with only a modest increase in current observed for the longer soaking times. This difference might be due to an increase in the surface area of Ir accessible to the electrolyte or the formation of iridium oxide due to prior electrochemical testing at expanded limits. In either case, some increase in C_{app} via either mechanism is anticipated and, overall, this site has exhibited a stable electrochemical response for more than one year of soaking in 0.1M PBS.

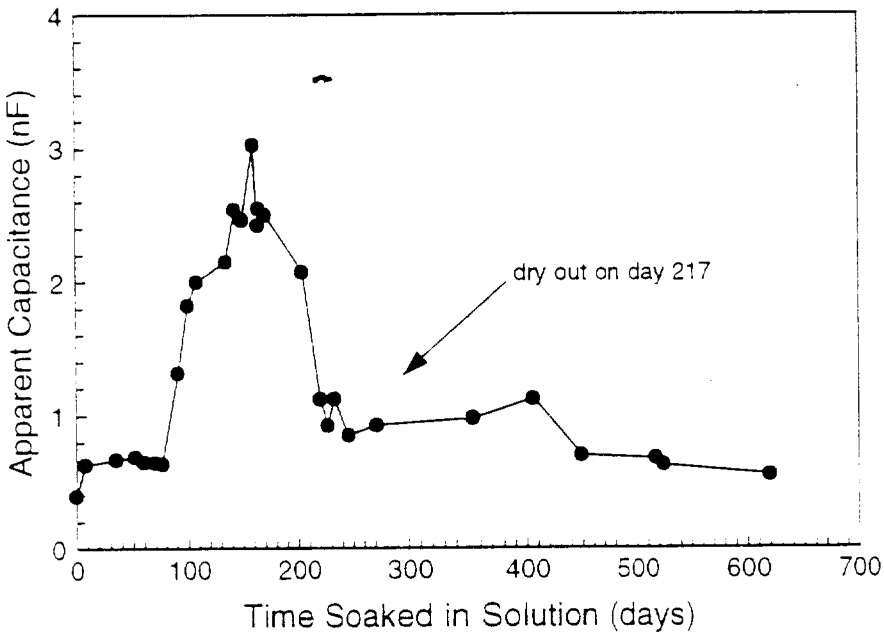

Figure 2-2 Apparent capacitance of site 1 on U. Michigan ribbon-cable probe No. 1 at 200 V/s for the 29 scan rate studies.

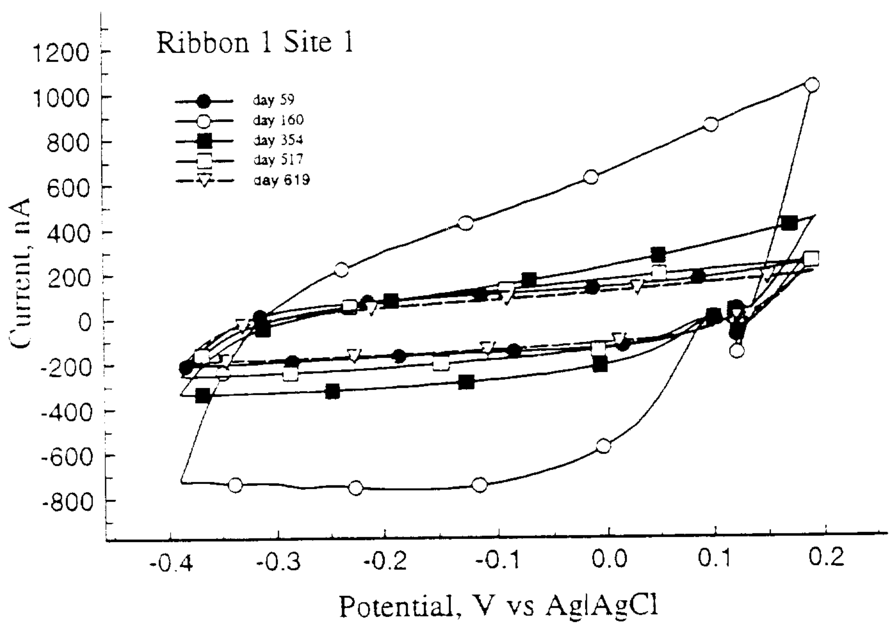

Figure 2-3 Voltammetric scans of site 1 on U. Michigan ribbon cable probe No.1 after 59, 160, 354, 517 and 619 days of soaking. Scan rate = 0.05 V/sec.

3. IRIDIUM ACTIVATION AND CHARGE INJECTION STUDIES

3.1. Experimental Setup

Phosphate-buffered saline (PBS), pH 7.3, is used for activation of the iridium electrode as well as pulse stimulation and evaluation of AIROF following the determination of charge injection limits. This electrolyte is deoxygenated by a flow of Ar gas. Potentials are measured against a silver-silver chloride reference electrode (Bioanalytical Systems, Inc.), No. MF-2074. One iridium electrode was used for this portion of the study: an iridium disk fabricated at EIC by casting 1 mm diameter wire in Epon resin 828 (ESA, 7.89 x 10^{-3} cm^2).

The electrochemical instrumentation used during activation and evaluation was an Amel 551 potentiostat modulated by a PAR 175 Universal Programmer. Cyclic voltammograms were recorded with a Bascom-Turner 5120 microprocessor controlled recorder and stored on floppy disks for analysis. During charge injection studies, the potential transients were monitored with a Tektronix 7623 oscilloscope.

The AIROF is formed by applying consecutive sets of 50 square-wave potential pulses between fixed cathodic and anodic limits, -0.70 and +0.80 V, using a 10 second halfwave until the desired charge storage capacity is achieved. After activation and after reaching the maximum charge injection level, cyclic voltammograms are acquired at scan rates of 0.05, 0.01 and 0.50 V/s between potential limits of -0.55 V and 0.75 V vs. Ag|AgCl. The anodic and cathodic charge storage capacities of the AIROF were determined by integrating the oxidation and reduction currents, respectively, after acquisition of the voltammograms. The unactivated Ir metal background was subtracted from the AIROF CV's before integration. Figure 3-1 illustrates a typical CV response of iridium activated in PBS using the above conditions for 450 pulses.

3.2. Influence of Charge Capacity on Charge Injection Limits

This quarter we began an investigation of the reproducibility of the charge injection process, the ability of AIROF to accommodate charge injection in the form of 200 µsec, biphasic, constant current pulses of equal cathodic and anodic magnitude for a sustained 3 minute period (4.5 E05 cycles). The maximum charge injection level is defined as the maximum charge density that can be applied during constant current pulsing without changing electrode behavior during cyclic voltammetry between specific potential limits for each electrode material.

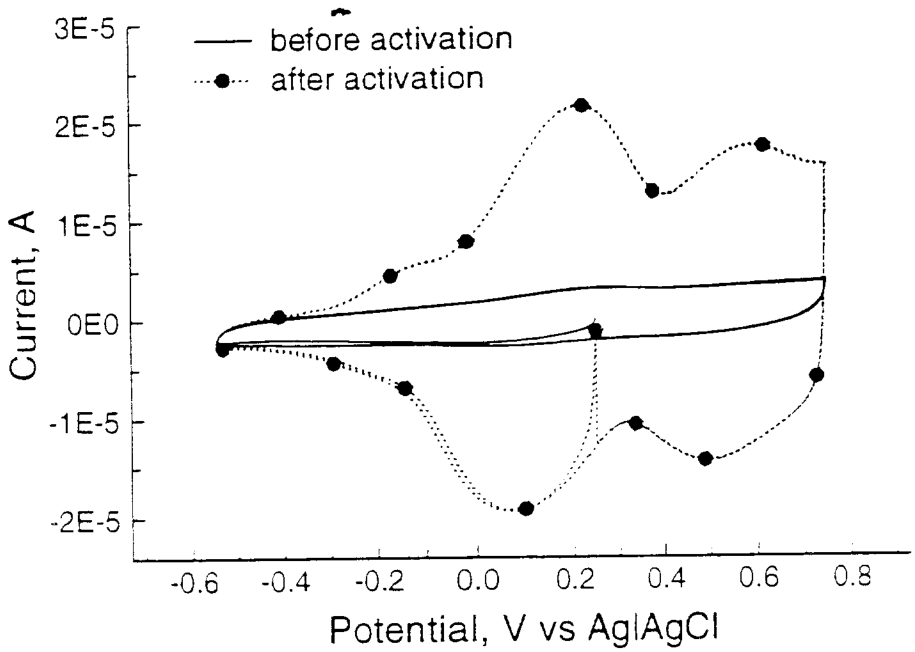

Figure 3-1 Iridium electrode at 0.05 V/s, before and after activation in 0.1M PBS. Cathodic charge storage capacity = 27.91 mC/cm^2. Anodic charge storage capacity = 27.83 mC/cm^2.

AIROF with three levels of cathodic charge storage capacity, 28, 37 and 47 mC/cm^2, were obtained at 0.05 V/sec in PBS. Completion of the constant current pulsing experiments resulted in maximum charge injection levels of 0.35, 0.30, and 0.12 mC/cm^2, respectively, as determined by the stability of cyclic voltammograms. Initially, only cathodic first pulses were recorded. Subsequently, cathodic first pulses were recorded, followed by anodic first pulses. The capability of the AIROF to accommodate constant current pulsing in PBS was assessed from the recorded potential transients and by comparison of cyclic voltammograms obtained in PBS before and after each incremental increase in pulse charge density. Figure 3-2 shows a series of electrochemical responses after progressively increasing the current density for constant current pulses of AIROF with a cathodic charge storage capacity of 47.25 mC/cm^2. After pulsing at 0.65 A/cm^2, the final cyclic voltammogram shows the typical distorted electrochemical response of AIROF which has changed due to pulsing conditions.

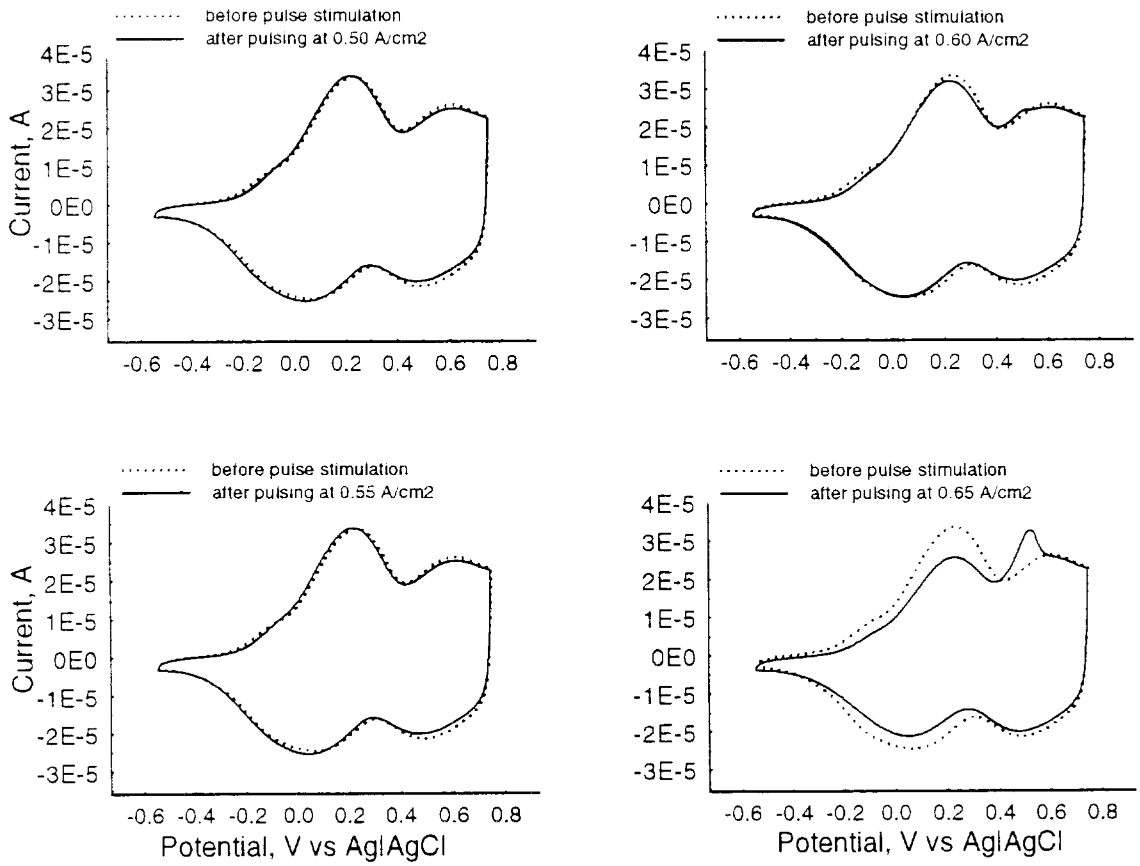

Figure 3.2 Series of electrochemical responses in PBS at 0.05 V/sec before and after incrementally increasing the pulse charge density for pulse stimulation. The AIROF has a cathodic charge storage capacity of 47.25 mC/cm^2 prior to constant current pulsing.

Figure 3-3 shows the relationship between charge storage capacity and the charge injection levels for the three pulsing experiments performed to date. The open symbols indicate the sequential charge injection levels at which the electrode was pulsed prior to reaching the maximum charge injection level. The solid symbols indicate the maximum charge injection level. The next highest charge injection level (not pictured) would show a cyclic voltammetric response indicating a change in electrochemical behavior. The data indicate that as charge storage capacity increases, the maximum charge injection level decreases.

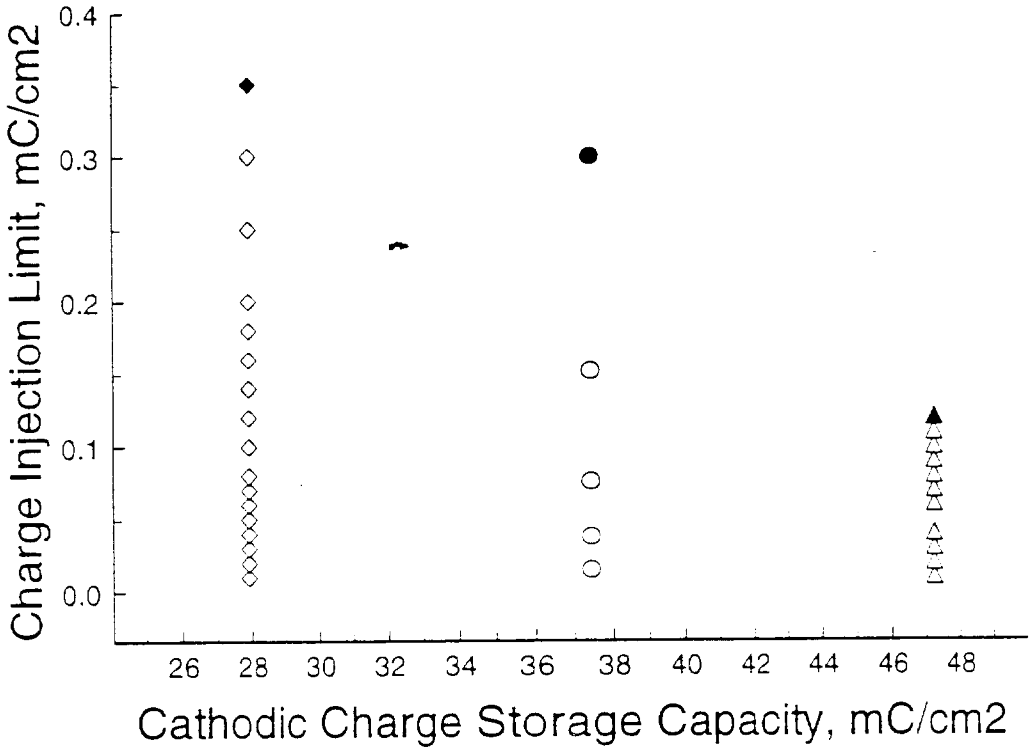

Figure 3.3 Charge injection limits of AIROF as a function of charge storage capacity at 0.05 V/sec. Solid symbols indicate maximum charge injection level at which electrochemical response remains unchanged. Open symbols are charge pulse injection levels used prior to reaching the maximum charge injection level.

4. FUTURE WORK

The evaluation of the reproducibility of maximum charge injection levels for AIROF in PBS will continue in the next quarter. The affect of activation parameters on charge injection and utilization of AIROF in PBS will be evaluated. The evaluation of charge injection levels for AIROF in 0.3M Na_2HPO_4 and for Ti:Ir graded interface electrodes will be undertaken in the next reporting period.